

Table 2

Ex No	Ester substituent	Route	α , β ratio	%Y	MP (°C)
2.1	Benzoyl	3A	4% α , 96% β	100	79
2.2	Benzoyl	3B	82% α , 18% β	93	85
2.3	Ethanoyl	3C	38% α , 62% β	94	77
2.4	Ethanoyl	3A	59% α , 41% β	98	87
2.5	Ethanoyl	3B	95% α , 5% β	86	102
2.6	Benzoyl	3A	5% α , 95% β	100	85
2.7	2-Naphthoyl	3A	3% α , 97% β	100	84
2.8	2-Naphthoyl	3B	94% α , 6% β	100	80
2.9	4-Biphenoyl	3A	8% α , 92% β	100	82
2.10	Cyclohexanoyl	3A	5% α , 95% β	99	77
2.11	Hexanoyl	3A	28% α , 72% β	100	80
2.12	n-Hexadecanoyl	3A	17% α , 83% β	100	65
2.13	n-Hexadecanoyl	3B	97% α , 3% β	100	52

5 Cellobiose octanonanoate, cellobiose octadecanoate and cellobiose heptanonaate reference materials, which are employed in Examples 5 to 17 hereinbelow.

Table 3

Ref	Acyl Groups	α , β ratio	%Y	MP (°C)
REF1	Nonanoyl	100% α ,	100	97
REF2	Nonanoyl	88% α , 12% β	98	80
REF3	Nonanoyl	1% α , 99% β	100	80
REF4	Decanoyl	85% α , 15% β	84	85
REF5	Nonanoyl	50% α , 50% β	0	114
REF6	Decanoyl	50% α , 50% β	0	105

Example 3

In this Example, further esters were made comprising
5 cellobiose heptanonanoate and a different ester group at the
anomeric position. The process comprised a variation of the
three stage process described in Example 1 above, the
principal differences being indicated herein.

- 10 In stage 1, a base catalyst was employed, producing β -D-
cellobiose octanonanoate.

D-(+)cellobiose (ACROS; 99% HPLC; 67 % β anomer), nonanoyl
acid chloride (83g, 0.47 mol), 96% ALDRICH, (assay; GC:
15 97.4% with remainder of 2-methyloctanoyl chloride); dry
chloroform 99+% (Sure Seal™ bottle, reagent grade, ALDRICH),
dry pyridine 99.8+% (sure seal bottle, reagent grade
ALDRICH), tetrahydrofuran (Fisher, reagent grade) and
methanol (FISHER, reagent grade) were used as received.

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A 250 ml three round bottom flask was fitted with a double surface condenser, a pressure equalising funnel and an overhead stirrer. All glassware had previously been dried overnight at 105°C.

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Clear, very faintly yellow nonanoyl chloride (83.2g, 0.47 mol) was added dropwise slowly, taking between 15 and 30 minutes with constant stirring to a solution of cellobiose (10g, 0.029 mol) in dry chloroform (40 mls) and dry pyridine (20 mls) at 50°C. The resultant heterogeneous pale yellow mixture was allowed to react for at 50°C under inert atmosphere and vigorous stirring until monitoring via proton NMR and HPLC indicated that no hepta-substituted cellobiose was present, a period of about 20 hours.

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The heterogeneous light yellow mixture was cooled down to room temperature. Then, the reaction mixture was poured into methanol (1000 mls) and stirred for about 15 minutes, producing a precipitate which was recovered by filtration, washed with 50 mls fresh methanol and dried in a vacuum oven (0.8 mbar, 80 Pa) at 40 to 45°C for 5 hours,. 39 g of a white solid was recovered.

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The white solid was recrystallised three times from a tetrahydrofuran/methanol mixture (75/200mls), filtered, washed with 50 ml of methanol and dried in a vacuum oven for 5 hours at 40-45°C. 18 g of a white solid (42%) was obtained.